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THE CHEMISTRY OF SILK

Silk is one of the most important fabrics in use at the present time. It is more widely used than any other fibre, not only because it is the strongest fibre known to Science but it is also proof against decay caused by dampness.

The varied uses as well as the different wearing qualities of silks has lead us to investigate some 51 samples of the more common silks, which were obtained from stores in Seattle and Tacoma.

In making these investigations the HNO_3 test proved to be the best for the qualitative study of silks, and the burning test for finding the amount of inorganic material present.

The results obtained from these test have been tabulated in comparison charts.

Before taking up the study of the results obtained from laboratory work, it is desirable to familarize ones self with the history and manufacturing processes of silk.

The history of silk begins about 1700 B. C. Hoang Ti, the third Emperor of China, who charged his wife to examine the silk worms and to test the practicability of using the thread from the cucoons. In her zeal she collected large numbers of the worms; fed them and discovered how to reel the silk and to make it into garments. She was deified and has been known as the "Godess of Silkworms."

The Chinese kept their method of obtaining the silk, a profound secret for nearly 2000 years. They sold the silk to the Persians, who for 1000 years carried the silk to Western Nations without knowing how or from what it was made.

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Aristotle was the first European to learn the true origin of the wrought silk. At this time, Pamphile in the Island of the Cos was importing raw silk from Persia, and, unraveling it, wove a silken gauze, which from its transparency, was called "Woven Wind." Because of its beautiful texture it was introduced into the courts.

In the 6th Century A. D. all the raw silk was still being imported from China by way of Persia. But when the Emperor Justinian engaged in war with that country, his supply of silk was cut off. However two Nestorian monks concealed silk worm eggs and brot them to Greece in 555 A. D.

The industry now spread over Greece and Syria and in 711 A. D. it was introduced in Spain. During the 16th Century the silk industry gained prominence in Italy.

Silk culture started in America in 1622, when James I, sent silk worm eggs, mulberry trees and printed instructions to Virginia, but the attempt was unsuccessful. In 1635, 3 pounds of silk were exported from Georgia. In 1658, 700 pounds were exported and 10,000 pounds (\$75,00) in 1754.

Connecticut began to rear silk worms in 1660 and for 84 years, this state lead all others in the amount of silk produced. During the Revolution the industry discontinued and was not revived until 1826, when a most determined effort was made to place silk growing on a paying basis. For ten years all went well. Then an attempt was made to supplant the white mulberry by the Chinese mulberry. A sudden infatuation seized the people; speculation began and prices advanced far beyond their real value. In the crisis of 1839, the bubble burst, bring ruin to thousands of people. Mulberry twigs which had been worth nearly their weight in gold could not be sold for ten cents a hundred

The company at Florence gave up the silk industry. However the Corticelli Company, which was started in 1838, is today noted for its silk.

Let us turn now to a study of the insect, which produces the silk. It is the larva of a small moth called *Sericana mori*. This moth is classed with the Lepidoptera or scaly winged insects, family — Bombycides or spinners. This specie of catter-pillar is commonly called the mulberry silk worm.

The silk worm has become domesticated, which has increased its silk producing capacity. It has lost ~~its~~ power of flight and changed to a white color.

The silk worm exists in fore stages, the egg, larva, chrysalis and adult.

The egg of the moth is nearly round, slightly flattened and closely resembles a turnip seed. When first laid it is yellow, but soon turns a gray or slate color if unfertilized.

The worm hatches in June. It is black in color, $\frac{1}{8}$ in. long and covered with long hair. It has a shiny nose and sixteen small legs. The jaws of a silk worm move side wise. They breath through spiracles, small holes which look like black spots on the sides of the body. The worms are velvety smooth and cold to the touch. The flesh is firm almost hard. The blood runs toward the head.

At all ages and times the worm secrets silk to protect itself from injury. After gaining its full growth the worm is ready to spin its cocoon. It loses its appetite and shrinks nearly an inch in length. The worm now seeks a resting place, while in this resting place the worm excretes silk in two long convoluted vessels, between the

forelegs and head, one on each side of the elementary canal. These vessels grow more slender and finally unite within the spinneret, from which the silk issues in a glutinous state and apparently in a single thread. The gummy liquid which unites the two strands hardens immediately when exposed to air. The silk is forced out by the contraction of the body.

The cocoon is now tough and compact, composed of a firm, continuous thread, wound in short figure eight loops. When the worm has finished spinning it is $1\frac{1}{4}$ in. long. Two days later by a final moult, its dried up skin breaks at the nose and is crowded back, revealing the third stage, the chrysalis. This is an oval cone, one inch in length. It is light yellow and immediately after moulting is soft to the touch. The fore legs have disappeared and in their place the four wings of the future moth are folded over the breast, together with six legs and two feelers. It soon turns brown and toughens into a hard shell. After two or three weeks, the chrysalis bursts. The moth ejects against the end of the cocoon, a strong alkaline liquid which dissolves the hard gummy lining. Then comes the adult moth.

The escape of the adult moth breaks so many threads, that the cocoons are ruined for reeling and so when ten days old, all those not intended, for reproducing, are placed in a steam heater. In Japan, the usual method is to place them in hot water. This deadens the chrysalis and the silk may then be reeled at any future time.

The cocoons, intended for the making of silk, are assorted as to color and texture. The loose silk from the outside is removed and the cocoons are placed in warm water to soften the gum. They are brushed and cleaned. The loose silk is taken off until each cocoon, shows but one thread. Then, two or more cocoons according to the size of thread

desired are placed in a basin of warm water and the threads are brot out together. The next process is the reeling of the silk.

Silk reeled by hand or foot power is known as "Re reel" silk. Silk reeled by power machinery is called "filature."

The skeins are now, assorted and put in canvass bags and soaked in warm soap suds over night. This softens the natural gum in the silk. The skeins are assorted and hung across poles in a steam heated room. The silk is then wound on three inch bobbins. In the doubling frames the thread from several bobbins is wound together. Then the spinner draws the thread from the bobbins upward to another bobbin. This spins the several strands brot together by the doubling process into one thread. In the twisting machine the silk is twisted in the opposite direction from the spinning. In the water stretcher the silk is wound on to the lower of two copper rolls. From the lower roll it passes upward to the upper roll, which turns faster than the lower one, there by stretching the silk. Then the silk is reeled into hanks.

Reeled silk is harsh and wiry as it contains considerable gum or seracin and it may also have much color in it.

Stripping or boiling off the gum, greatly reduces the weight of the silk for 25% or more is often lost. The result is a silk of a duller appearance and a firmer touch, suitable for gros grains. "Ecrú" silk has less gum removed, probably from 2 to 5%. A bath of dilute acetic acid is given if "scroop" the crunching sound of silk is desired. This is found in taffetas to a great extent.

The practice, of weighting, the silk, may begin with the raw material. Hydrosopic mineral salts and fatty and petroleum oils are sometimes used in the process of reeling, in connection with or in place of the weak soapy solution, which is the only necessary aid in softening the cucoons.

The price of raw silk averages about \$5.00 a pound, consequently weighting is used to make up for the loss of weight by boiling off the silk.

The substances used in dyeing which add weight to silk, do not always contain salts of heavy metals, when they do it is often a tin-phosphate-silicate. Successive baths of stannic chloride, sodium phosphate and sodium silicate are used and the amount of weighting is determined accurately, by the number of dips and the time allowed to each dip.

Goods weighted with tin compounds are especially liable to deterioration, on exposure to air and light. This is likely due to the hydrolytic effect of moisture on the stannic chloride, which liberating hydrochloric acid, acts as a solvent upon the silk.

Aluminum sulfate is sometimes added to tin compounds to give a heavier thread. Sugar is often used as a weighting. It is applied after the silk is dyed. Each dip in the sugar solution may add 10% in weight. Lead acetate or more often pink salt which is the double salt of stannic and ammonium chloride, is often used.

Black silks may be "pure dye", with iron tannate as the mordant or they may be "dynamited", that is especially heavy loaded. Logwood is still preferred to coal tar colors for black silks. For this method, after boiling off, the silk is steeped in a bath of iron nitrate and passed into a soap and iron nitrate bath, which adds a given weight of iron soap. The skein is then passed into a bath of potassium ferrocyanide, which with the iron present, produces Prussian Blue. This is followed by a bath of cutch. Tannin absorbed from the cutch increases the weight 35 to 40% and here stannous chloride may be added to bring

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it up to any desired weight. The silk is now mordanted in an iron bath and the dyeing finished in a log wood solution.

Piece dyed goods such as liberty silks, foulards, chiffons and shatungs were formerly little weighted in this country, but the practice of adding some weighting to these is increasing.

Silk is dyed in a neutral, alkaline or slightly acidified bath to which in practice soap and boiled off liquor are usually added. An after treatment with a tannic acid bath followed by rinsing in a solution of tartar emetic, improves the fastness of the color.

Following the preparation ~~of~~ the silk thread, comes the weaving and the designing of the silk. The silk designer requires years of training and artistic taste to weave silks in the right way. Standard silks, which include the following: ^{glaze silks} is the name given to silk with contrasting colors of warp and woof; pongee and shatung; taffeta and crepe de chene. In twills are the surah and others such as serge and foulard. In ribs and cords there are the ottoman gros grain, poplin and terry velvets. In the satin weaves are the messaline, satin and peau de cygne. In gauze weaving are the marquissettes; in jacquard, the brocade and tie silks.

After the weaving and dyeing of the silk, a finish is given to it to increase the lustre. Finishings are of two kinds, mechanical ~~of~~ by dressings. The first includes, calendering, mangling or pressing. Dressings either soften, stiffen, harden, glaze, strengthen or water-proof the silk. The following dressings are in constant use, dextrin, rice water, isinglass, gelatin, starch, glues, gums and waxes. The waxes are dissolved in an alkali and the gums are softened with castor oil. Pure silk requires no dressings. It is used only in the poorer cheaper silks to add to their body.

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For some purposes, it is not necessary to use true silk, consequently many artificial silks have been manufactured. These materials give the same general effect as silk but of course are not as durable. However in some stores we find artificial silks, sold under the name of true silks.

Nearly all artificial silks are made by treating cellulose in some way, to produce filaments of great beauty. These filaments lack the softness and elasticity of true silk and are liable to disintegrate on washing. The principal artificial silks on the markets are the nitrocellulose, cuprate and viscose silks. They are known as lustracellulose, from their cellulose origin. The solutions are forced thru small capillary tubes to give the shape of threads.

Tulle or maline, instead of being forced thru capillary tubes, is pressed upon an engraved cylinder and at the same time, receives the hardening treatment.

The following charts are results of work done in the laboratory. To estimate the amount of weighting of the amount of inorganic materials present in the silk.

A small piece of the silk was weighed very accurately and then heated in the presence of air until all the organic matter had been oxidized. The remaining ash, represented roughly the amount of inorganic weighting present.

In order to make a qualitative examination, the silk was treated with concentrated nitric acid, bring^{ing} the solution to a boil of it did not dissolve immediately.

CONCLUSIONS:

The results of burning 51 different samples of silk, show:

1. When fibres have been coated to excess the form of the fabric, will persist after the animal matter has been burned.
2. The following table shows the average weighting present in silks tested:

Taffetas-----	22. %
Crepe de chene-----	7. %
Georgette-----	9. %
Poplin-----	.2%
Satin-----	17.1%
Japan silks-----	8.1%
China silks----	no weighting
Messaline-----	19. %

3. Black brown and green silks seem to be weighted heavier than other colors.
4. The presence of tin compounds gives a characteristic white or gray ash.
5. The presence of iron compounds gives a characteristic reddish brown ash.
6. Aluminum sulfate made the fibres heavier than other weightings.
7. The most expensive silks usually contain but a small percent of weighting. However we find a few of the cheaper silks that are practically free from weighting.

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The results of treating 51 samples of silk with nitric acid show:

1. Silks containing wool take a gelatinous form, while cotton remains undissolved.
2. Silks containing weighting in the form of tin or aluminum sulfate remain undissolved.
5. Artificial silks remain in a solid mass.

#1 Tabulation

1	Taffeta	Brown	36	\$3.50	Stone-Fisher
2	Taffeta	Brown	36	3.50	Stone-Fisher
3	Crepe de Chine	Brown	36	1.69	Stone-Fisher
4	Crepe de Chine	Brown	36	1.69	Stone-Fisher
5	Polin	Blue	40	5.98	Rhodes
6	Georgette	Pink	40	1.98	Rhodes
7	Chiffon Taffeta	Brown	40	5.00	Rhodes
8	Messaline	Lavendar	36	1.89	Fair House
9	Crepe de Chine	White	36	2.50	Rhodes
10	Jap Silk (figured)	Blue, Mixture	36	2.98	Stone-Fisher
11	Taffeta (changeable)	Blue	36	2.25	Rhodes
12	Crepe de Chine	Brown	40	1.75	Rhodes
13	Georgette	Gray	40	3.50	Rhodes
14	Crepe de Chine	Yellow	40	2.00	Rhodes
15	Georgette	Pink	40	1.50	Jap Store
16	Surah	Plaid	36	3.5	Fair House
17	Georgette	Blue	40	3.00	Peoples
18	Satin, (Goetz)	Brown	36	2.50	Fair House
19	Taffeta	Blue	36	2.50	Rhodes
20	Crepe de Chine	Pink	40	1.29	Stone-Fisher
21	Satin	Brown	36	3.50	Fair House
22	Messaline	Orange	36	1.98	Fair House
23	China Silk	White	36	2.50	In China
24	Glase' silk	Pink, Blue	36	2.50	Feist-Bachrach
25	Satin	Black	36	2.40	Profitts-Chenai
26	Charmeuse, (Satin)	Blue	36	1.69	Stone Fisher
27	Pongee	Tan	36	.98	Feist-Bachrach
28	Poplin	Gray	36	1.50	Rhodes Seattle
29	Taffeta	Blue-Tan	36	3.00	Feist-Bachrach
30	Taffeta	Plaid	36	2.50	Rhodes
31	Silk mixture	Blue	36	1.25	Fair House
32	Messaline	Yellow	36	1.49	Sears Roebuck
33	Taffeta	Green	36	1.50	Sears Roebuck
34	Poplin	Blue	36	.98	Fair House
35	Jap Silk	White	36	.89	Fair House
36	Taffeta	Blue	36	1.69	Fair House
37	Crepe de Chine	White	36	1.29	Sears Roebuck
38	Taffeta	Yellow	36	3.50	Rhodes
39	Crepe de Chine	Red	35	1.49	Frazier-Patters
40	Peau de Soir	Black	36	2.25	Feist Bachrach
41	Satin	Black	36	4.50	Rhodes
42	Foulard	Blue	40	3.50	Feist Bachrach
43	Poplin	Yellow	35	1.00	Sears Roebuck
44	Taffeta	Blue	36	2.50	Feist Bachrach
45	China Silk	Gray-Blue	36	2.50	Rhodes
46	Gros de London	Green-Yellow	36	2.50	Peoples
47	Goetz Satin	Brown	36	2.50	Peoples
48	Figured Georgette	Blue-Pink	40	1.29	Stone Fisher
49	Chiffon	Green	36	.35	Sears Roebuck
50	Silk Stocking	Black		1.50	Peoples
51	Lining	Tan	36	.98	Rhodes

#2 Tabulation

1	% of weighting in Silk	Character of ash	Action with Nitric
1	.39%	Gray	Wool did not dissolved. Warp all dissolved.
2	.38%	Dark Gray	Warp all dissolved.
3	.19%		Dissolved quickly when warmed.
4	No ash.		Dissolved quickly when warmed.
5	.11%	Dark brown, metal like large granules.	Wool dissolved when heated. Warp form gelatinous (presence of wool)
6	No ash.		Dissolved when heated.
7	.13%		Dissolved quickly without heat.
8	.31%	Gray ash	When heated ppt. gelatinous.
9	.12%	Green, metal like, large granules.	Dissolved when heated.
10	8.9%		Dissolved when heated.
11	.47%		Dissolved when heated.
12	1%		Dissolved when heated.
13	No ash.		Dissolved when heated.
14	No ash.		Dissolved when heated, slowly.
15	.16%	White	Flaky ppt. when heated.
16	.71%	White-gray.	Flaky ppt. when heated.
17	.39%	White	Flaky ppt. when heated.
18	.57%	Gray.	Stayed in solid mass when boiled.
19	.50%	White.	Stayed in solid mass when boiled. (slow action.)
20	No ash.		Solid mass when heated.
21	No ash.		Dissolved when heated.
22	No ash.		Solid mass when heated.
23	No ash.		Dissolved when heated.
24	.28%	White.	Solid mass after heating
25	.3.2%	Orange.	When heated Ppt. (brown)
26	No ash.		Dissolved quickly when heated.
27	.17%	White.	Dissolved when heated.
28	No ash.		Warp did not dissolve, wool dissolved.
29	1.9%	White	Did not dissolve.
30	.36%	Gray-white.	Did not dissolve.
31	No ash.		Did not dissolve when heated.
32	.26%	Heavy white ash	Did not dissolve.
33	.20%	Heavy white ash	Did not dissolve.
34	No ash.		Warp did not dissolve, wool dissolved.
35	.7.4%	Fine gray ash.	Did not dissolve.

#3 Tabulation

No.	% of weighting in Silk	Character of ash	Action with Nitric Acid
36	.38%	White solid ash.	Wool did not dissolve
37	.43%	Gray ash.	Dissolved on heating
38	.16%	White solid ash.	Did not dissolve when heated.
39	No ash.		Dissolved on heating
40	.50%	Brown ash.	Dissolved on heating
41	.11%	Brown ash.	Dissolved on heating
43	No ash.		Dissolved quickly on heating.
45	No ash.		Did not dissolve when heated.
44	.11%	Gray ash.	Did not dissolve when heated.
45	No ash.		Dissolved quickly on heating.
46	.31%	Solid gray ash.	Did not dissolve.
47	.21%	Dark gray ash.	Did not dissolve when heated.
48	No ash.		Dissolved quickly with out heating.
49	No ash.		Dissolved quickly with out heating.
50	No ash.		Dissolved quickly with out heating.
51	.24%	Brown and white ash.	Warp did not dissolve Wool dissolved.

#4

COMPARSION TABLE

No.	% of weighting	\$	Price	Name
1	39%		3.50	Taffeta
2	38%		3.50	Taffeta
3	19%	3.50	1.69 (sale)	Crepe de chene
4	No ash	3.50	1.69 (sale)	Crepe de chene
5	11%		5.98	Poplin
6	No ash		1.98	Georgette
7	13%		5.00	Chiffon Taffeta
8	31%		1.89	Messalin
9	12%		3.50	Crepe de Chene
10	81%		3.95	Jap silk, figured.
11	47%		3.25	Taffeta
12	1%		1.75	Crepe de chene
13	No ash		3.50	Georgette
14	No ash		2.00	Crepe de Chene
15	16%		1.50	Georgette (Jap store)
16	71%		2.50	Surah silk
17	29%		3.00	Georgette
18	57%		3.50	Goetz satin
19	50%		2.50	Taffeta
20	No ash	2.00	1.29 (sale)	Crepe de Chene
21	No ash		3.50	Satin
22	No ash		1.98	Messaline
23	No ash		2.50	China silk
24	28%		2.50	Satin
25	3.2%		2.40	Satin
26	No ash	3.50	1.69 (sale)	Charmeuse
27	17%	1.25	.98 (sale)	Pongee
28	No ash		1.50	Poplin
29	1.9%		3.00	Taffeta
30	36%		2.50	Taffeta
31	No ash		1.25	Silk mixture
32	26%		1.49	Messaline
33	20%		1.50	Taffeta
34	No ash		.98	Poplin
35	7.4%		.89	Jap silk
36	38%		1.69	Taffeta
37	42%		1.29	Crepe de Chene
38	16%		3.50	Taffeta
39	No ash		1.49	Crepe de Chene
40	50%		2.25	Peau de Soir
41	11%		4.50	Satin
42	No ash		3.50	Foulard
43	No ash		1.00	Poplin
44	11%		2.50	Taffeta
45	No ash		2.50	China silk
46	31%		2.50	Gros de London
47	21%		2.50	Groetz satin
48	No ash	3.50	1.29 (sale)	Georgette
49	No ash		.39	Chiffon
50	No ash		1.50 pr. pair	Silk stockings
51	24%		.89	Lining

CONCLUSIONS

	Character of Weighting	Action with Hnq3. shows:
1	Sn	Sn in woof
2	Sn	Sn in woof
3	No ash	Pure silk
4	No ash	Pure silk
5	fe	Wool in warp
6	No ash	Pure silk
7	No ash	Pure silk
8	Unknown green metal ash	Weighting is soluble
9	Sn	Artificial silk
10	Unknown green metal ash	Weighting is soluble
11	No ash	Pure silk
12	No ash	Pure silk
13	No ash	Pure silk
14	No ash	Dressings
15	Sn	Cotton also Sn
16	Sn	Sn
17	Sn	Sn and cotton
18	Sn-al(SO ₄) ₃	Sn
19	Sn-al(SO ₄) ₃	Sn and dressings
20	No ash	Pure silk
21	No ash	Pure silk
22	No ash	Pure silk
23	No ash	Pure silk
24	Sn-al(SO ₄) ₃	Sn
25	Fe	Sn or Al
26	No ash	Pure silk
27	Sugar	Soluble weighting
28	No ash	Cotton in warp
29	Sn	Sn
30	Sn	Sn
31	No ash	Cotton
32	Sn	Sn
33	Sn	Sn
34	No ash	Cotton in warp
35	Sn	Sn
36	Sn-al(SO ₄) ₃	Sn
37	Unknow sol. weighting	Soluble weighting
38	Sn	Sn
39	No ash	Pure silk
40	Fe	Sn or Al
41	Fe	Soluble weighting Fe
42	No ash	Pure silk
43	No ash	Cotton
44	Sn	Sn
45	No ash	Pure Silk
46	Sn-Al(SO ₄) ₃	Sn and Al
47	Sn	Sn
48	No ash	Pure silk
49	No ash	Pure silk
50	no ash	Pure silk
51	Sn and Fe	Cotton in warp